

PCT

INTERNATIONAL SEARCH REPORT

(PCT Article 18 and Rules 43 and 44)

Applicant's or agent's file reference P18142A/BOU	FOR FURTHER ACTION see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below.	
International application No. PCT/GB 98/ 01597	International filing date (day/month/year) 01/06/1998	(Earliest) Priority Date (day/month/year) 31/05/1997
Applicant GILTECH LIMITED et al.		

This International Search Report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This International Search Report consists of a total of 3 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. ☐ **Certain claims were found unsearchable**(see Box I).

2. ☐ **Unity of invention is lacking**(see Box II).

3. ☐ The international application contains disclosure of a **nucleotide and/or amino acid sequence listing** and the international search was carried out on the basis of the sequence listing

☐ filed with the international application.

☐ furnished by the applicant separately from the international application,

☐ but not accompanied by a statement to the effect that it did not include matter going beyond the disclosure in the international application as filed.

☐ Transcribed by this Authority

4. With regard to the **title**, ☒ the text is approved as submitted by the applicant

☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,

☐ the text is approved as submitted by the applicant

☒ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this International Search Report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is:

Figure No. ---- ☐ as suggested by the applicant.

☐ None of the figures.

☐ because the applicant failed to suggest a figure.

☐ because this figure better characterizes the invention.

Box III TEXT OF THE ABSTRACT (Continuation of item 5 of the first sheet)

There is provided a method for forming a water-soluble glass fibre or wool. The method comprises heating the glass composition above its melting point to produce a molten glass and then cooling the molten glass slowly to a pre-selected working temperature at which the fibres will be drawn. Suitable working temperature include those in a range of 400 to 1000° C. The working temperature will usually be at least 200°C lower than the temperature to which the molten glass is heated above its melting point and may be 50-300°C above the T_g of the glass. Phosphorous pentoxide is suitable as a glass former and B₂O₃ may be present as an additive. Optionally the glass may release silver ions, e.g. by addition of silver orthophosphate during manufacture of the glass.

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 98/01597

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C03C13/00 C03B37/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C03B C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 578 023 A (CORNING INC) 12 January 1994 see page 4, line 20 - page 5, line 34; claim 4; tables I-III ---	1-6
A	WO 86 04807 A (UNIVERSITY OF DAYTON, US) 28 August 1986 cited in the application see claims 1-5 ---	1
A	WO 92 07801 A (ZIMMER INC) 14 May 1992 cited in the application see claims 1,7,8 ---	1
	--- -/--	



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

° Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search

21 August 1998

Date of mailing of the international search report

02/09/1998

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Stroud, J

INTERNATIONAL SEARCH REPORT

International Application No

PCT/GB 98/01597

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>DATABASE WPI Section Ch, Week 08 Derwent Publications Ltd., London, GB; Class D22, AN 83-18194K XP002075169 & JP 58 004 821 A (MITSUBISHI MINING & CEMENT CO) , 12 January 1983 see abstract</p> <p>---</p>	1
A	<p>DATABASE WPI Section Ch, Week 25 Derwent Publications Ltd., London, GB; Class L01, AN 73-80941U XP002075170 & JP 48 042 814 B (NIPPON SHEET GLASS CO LTD) , 1973 see abstract</p> <p>-----</p>	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/GB 98/01597

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
EP 0578023	A	12-01-1994	US 5330940 A CA 2084471 A JP 6056472 A	19-07-1994 07-01-1994 01-03-1994
WO 8604807	A	28-08-1986	US 4604097 A AU 5518386 A EP 0211942 A JP 7005335 B JP 62501905 T	05-08-1986 10-09-1986 04-03-1987 25-01-1995 30-07-1987
WO 9207801	A	14-05-1992	US 5215563 A AU 658380 B AU 8952991 A CA 2094931 A EP 0555390 A JP 6504754 T	01-06-1993 13-04-1995 26-05-1992 02-05-1992 18-08-1993 02-06-1994

PCT COOPERATION TREATY

PCT

NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

United States Patent and Trademark
Office
(Box PCT)
Crystal Plaza 2
Washington, DC 20231
ÉTATS-UNIS D'AMÉRIQUE

in its capacity as elected Office

Date of mailing (day/month/year) 14 January 1999 (14.01.99)	
International application No. PCT/GB98/01597	Applicant's or agent's file reference P18142A/BOU
International filing date (day/month/year) 01 June 1998 (01.06.98)	Priority date (day/month/year) 31 May 1997 (31.05.97)
Applicant GILCHRIST, Thomas et al	

1. The designated Office is hereby notified of its election made:

☒ in the demand filed with the International Preliminary Examining Authority on:
21 December 1998 (21.12.98)

☐ in a notice effecting later election filed with the International Bureau on:

2. The election ☒ was
☐ was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

<p>The International Bureau of WIPO 34, chemin des Colombettes 1211 Geneva 20, Switzerland</p> <p>Facsimile No.: (41-22) 740.14.35</p>	<p>Authorized officer</p> <p>Nicola Wolff</p> <p>Telephone No.: (41-22) 338.83.38</p>
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INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P18142A/PKE/BOU	FOR FURTHER ACTION See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/GB98/01597	International filing date (day/month/year) 01/06/1998	Priority date (day/month/year) 31/05/1997
International Patent Classification (IPC) or national classification and IPC C03C13/00		
Applicant GILTECH LIMITED et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 4 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☐ Certain observations on the international application

Date of submission of the demand 21/12/1998	Date of completion of this report - 6. 09. 99
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Ritter, R Telephone No. +49 89 2399 8578 

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB98/01597

I. Basis of the report

1. This report has been drawn on the basis of (*substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments.*).

Description, pages:

1-17 as originally filed

Claims, No.:

1-10 as originally filed

2. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
☐ the claims, Nos.:
☐ the drawings, sheets:

3. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

4. Additional observations, if necessary:

V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. Statement

Novelty (N)	Yes:	Claims	9,10 yes
	No:	Claims	1-8 no
Inventive step (IS)	Yes:	Claims	9,10 yes
	No:	Claims	1-8 no
Industrial applicability (IA)	Yes:	Claims	1-10 yes
	No:	Claims	

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT**

International application No. PCT/GB98/01597

2. Citations and explanations

see separate sheet

**INTERNATIONAL PRELIMINARY
EXAMINATION REPORT - SEPARATE SHEET**

International application No. PCT/GB98/01597

1) Reference is made to the following documents:

D1: ~~EP-A-0578023~~

D2: WO 86 04807 (cited in the application page 3, line 5)

D3: WO 9207801 (cited in the application page 3, line 22).

2) The present application does not fulfil the requirements of Article 33(2) PCT:
The subject-matter of claims 1-8 of the present application is known from
D1 (page 2, lines 49-56; page 4, line 20 -page 5, line 34; claim 4; tables I-III).
Also the subject-matter of D2 (claims 1-5) and D3 (claims 1,7,8) is novelty destroying
for claim 1 of the present application.

3) None of the cited documents discloses or suggests a method wherein a silver ion
releasing glass is manufactured (claims 9, 10 of the present application).

The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:

IPEA/ US

PCT

CHAPTER II

DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty and hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only

Identification of IPEA		Date of receipt of DEMAND	
Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION		Applicant's or agent's file reference LE9-99-029	
International application No.	International filing date (day/month/year) 26 October 2000 (26.10.00)	(Earliest) Priority date (day/month/year) 29 October 1999 (29.10.99)	
Title of invention. INK COMPOSITIONS CONTAINING ULTRAVIOLET ABSORBERS			
Box No. II APPLICANT(S)			
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) Lexmark International, Inc. IP Law Dept. 740 West New Circle Road Lexington, KY 40550 US		Telephone No.: 859-232-7843 Facsimile No.: 859-232-7850 Teleprinter No.:	
State (that is, country) of nationality: US		State (that is, country) of residence: US	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)		RECEIVED JUN - 4 2001 TC 1700 MAIL ROOM	
State (that is, country) of nationality:		State (that is, country) of residence:	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)			
State (that is, country) of nationality:		State (that is, country) of residence:	
<input type="checkbox"/> Further applicants are indicated on a continuation sheet.			

Sheet No. 2

International application No.

Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE

The following person is ☒ agent ☐ common representative
 and ☒ has been appointed earlier and represents the applicant(s) also for international preliminary examination.
☐ is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.
☐ is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

DASPIIT, Jacqueline M.
 Lexmark International, Inc.
 Intellectual Property Law Dept.
 740 West New Circle Road
 Lexington, KY 40550
 US

Telephone No.:

859-232-7843

Facsimile No.:

859-232-7850

Teleprinter No.:

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION

Statement concerning amendments:*

1. The applicant wishes the international preliminary examination to start on the basis of:

☒ the international application as originally filed
 the description ☒ as originally filed
☐ as amended under Article 34

the claims ☒ as originally filed
☐ as amended under Article 19 (together with any accompanying statement)
☐ as amended under Article 34

the drawings ☒ as originally filed
☐ as amended under Article 34

2. ☐ The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.3. ☐ The applicant wishes the start of the international preliminary examination to be postponed until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

Language for the purposes of international preliminary examination:

English

☒ which is the language in which the international application was filed.
☐ which is the language of a translation furnished for the purposes of international search.
☐ which is the language of publication of the international application.
☐ which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

Box No. V ELECTION OF STATES

The applicant hereby elects all eligible States (that is, all States which have been designated and which are bound by Chapter II of the PCT)

excluding the following States which the applicant wishes not to elect:

Sheet No. 3

International application No.

Box No. VI CHECK LIST

The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination:

- | | | | |
|--|---|---|--------|
| 1. translation of international application | : | 0 | sheets |
| 2. amendments under Article 34 | : | 0 | sheets |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | 0 | sheets |
| 4. copy (or, where required, translation) of statement under Article 19 | : | 0 | sheets |
| 5. letter | : | 0 | sheets |
| 6. other (specify) | : | 0 | sheets |

For International Preliminary Examining Authority use only

received not received

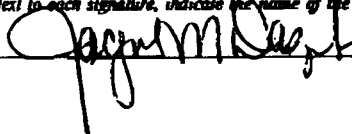
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>
<input type="checkbox"/>	<input type="checkbox"/>

The demand is also accompanied by the item(s) marked below:

- | | |
|---|---|
| 1. <input checked="" type="checkbox"/> fee calculation sheet | 4. <input type="checkbox"/> statement explaining lack of signature |
| 2. <input type="checkbox"/> separate signed power of attorney | 5. <input type="checkbox"/> nucleotide and or amino acid sequence listing in computer readable form |
| 3. <input checked="" type="checkbox"/> copy of general power of attorney; reference number, if any: | 6. <input type="checkbox"/> other (specify): |

Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).



Jacqueline M. Daspit, Agent

For International Preliminary Examining Authority use only

- Date of actual receipt of DEMAND:
- Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):
- ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply.
 ☐ The applicant has been informed accordingly.
- ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.
- ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

For International Bureau use only

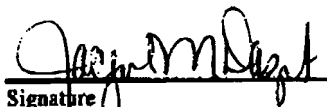
Demand received from IPEA on:

CHAPTER II

PCT

FEE CALCULATION SHEET

Annex to the Demand for international preliminary examination

International application No. <hr/> Applicant's or agent's file reference LE9-99-029	For International Preliminary Examining Authority use only <hr/> Date stamp of the IPEA
Applicant LEXMARK INTERNATIONAL, INC.	
Calculation of prescribed fees	
1. Preliminary examination fee	<div style="border: 1px solid black; padding: 2px; display: inline-block;">490.00</div> <div style="border: 1px solid black; padding: 2px; display: inline-block; margin-left: 10px;">P</div>
2. Handling fee (<i>Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.</i>)	<div style="border: 1px solid black; padding: 2px; display: inline-block;">153.00</div> <div style="border: 1px solid black; padding: 2px; display: inline-block; margin-left: 10px;">H</div>
3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box.....	<div style="border: 1px solid black; padding: 5px; display: inline-block;"> <div style="display: flex; justify-content: space-between;"> USD 643.00 </div> <div style="text-align: center; border-top: 1px solid black; padding-top: 5px;">TOTAL</div> </div>
Mode of Payment	
<input checked="" type="checkbox"/> authorization to charge deposit account with the IPEA (see below)	<input type="checkbox"/> cash
<input type="checkbox"/> cheque	<input type="checkbox"/> revenue stamps
<input type="checkbox"/> postal money order	<input type="checkbox"/> coupons
<input type="checkbox"/> bank draft	<input type="checkbox"/> other (specify):
Deposit Account Authorization (<i>this mode of payment may not be available at all IPEAs</i>) The IPEA/ <u>US</u> <input checked="" type="checkbox"/> is hereby authorized to charge the total fees indicated above to my deposit account. <input checked="" type="checkbox"/> (<i>this check-box may be marked only if the conditions for deposit accounts of the IPEA so permit</i>) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account.	
12-1213 Deposit Account Number	<div style="text-align: right;">  Signature </div> <div style="text-align: left;"> <div style="border-bottom: 1px solid black; display: inline-block; width: 150px;">29 May 2001</div> Date (day/month/year) </div>

PCT**GENERAL POWER OF ATTORNEY***(for several international applications filed under the Patent Cooperation Treaty)*

(PCT Rule 90.5)

The undersigned person(s)*(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

Lexmark International, Inc.
740 West New Circle Road
Lexington, KY 40550
US

hereby appoints (appoint) the following person as:

☒ agent☐ common representative**Name and address***(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

MCARDLE, John J.; PEZDEK, John V.; BRADY, John A.; LAMBERT, D. Brent;
SANDERSON, Michael T.; DASPIT, Jacqueline M.; MORGAN, Deborah E.; JACOBS,
Elizabeth C.

Lexmark International, Inc.
740 West New Circle Road
Lexington, KY 40550
US

to represent the undersigned before

☒

all the competent International Authorities

☐

the International Searching Authority only

☐

the International Preliminary Examining Authority only

in connection with any and all international applications filed by the undersigned with the following Office

RO/US

as receiving Office

and to make or receive payments on behalf of the undersigned.

Signatures of the applicant(s) *(where there are several persons, each of them must sign; next to the signature, indicate the name of the person signing and the capacity in which the person signs, if such capacity is not obvious from reading this power):*

Vincent J. Cole, Vice President, General Counsel and Secretary

V. J. Cole

Date:

5-15-01

PCTWORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C03C 13/00, C03B 37/02	A1	(11) International Publication Number: WO 98/54104 (43) International Publication Date: 3 December 1998 (03.12.98)
(21) International Application Number: PCT/GB98/01597 (22) International Filing Date: 1 June 1998 (01.06.98) (30) Priority Data: 9711178.5 31 May 1997 (31.05.97) GB (71) Applicant (for all designated States except US): GILTECH LIMITED [GB/GB]; 12 North Harbour Estate, Ayr KA8 8AA (GB). (72) Inventors; and (75) Inventors/Applicants (for US only): GILCHRIST, Thomas [GB/GB]; 67 Midton Road, Ayr KA7 2TW (GB). HEALY, David, Michael [IE/GB]; Midton House, By Alloway KA7 4EZ (GB). (74) Agent: OUZMAN, Beverley; Murgitroyd & Company, 373 Scotland Street, Glasgow G5 8QA (GB).		(81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GE, GH, GM, GW, HU, ID, IL, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: METHOD OF PRODUCING WATER-SOLUBLE GLASS FIBRES		
(57) Abstract <p>There is provided a method for forming a water-soluble glass fibre or wool. The method comprises heating the glass composition above its melting point to produce a molten glass and then cooling the molten glass slowly to a pre-selected working temperature at which the fibres will be drawn. Suitable working temperature include those in a range of 400 to 1000 °C. The working temperature will usually be at least 200 °C lower than the temperature to which the molten glass is heated above its melting point and may be 50-300 °C above the T_g of the glass. Phosphorous pentoxide is suitable as a glass former and B₂O₃ may be present as an additive. Optionally the glass may release silver ions, e.g. by addition of silver orthophosphate during manufacture of the glass.</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

1 **"Method of Producing Water-Soluble Glass Fibres"**

2

3 The present invention relates to a method for the
4 production of water soluble glass fibres and glass
5 wool.

6

7 It is known that certain glasses, in which the usual
8 glass former, silicon dioxide, is replaced with
9 phosphorous pentoxide, are soluble in water and body
10 fluids. The rate of dissolution is controlled largely
11 by the addition of glass modifiers such as calcium
12 oxide. In simple terms, the greater the concentration
13 of the modifier the slower the rate of dissolution.
14 The rate of dissolution may range from minutes through
15 to several years.

16

17 Soluble phosphate based glasses which have demonstrated
18 good biocompatibility can incorporate inorganic metals
19 such that a sustained release of the metals can be
20 provided at the wound site. Such materials can also
21 find use in mechanical applications where, for example,
22 slow release of an anti-corrosion agent may be
23 beneficial.

24

25 Certain applications require that the glass is in the

1 form of wool or fibres for mechanical applications such
2 as insulation wool and packaging. Thus, for example,
3 Mohr et al in "Fibre Glass" (Van Norstrand, Reinhold
4 Company, New York 1978) and Jaray in "A New Method of
5 Spinning Glass Fibres" (28th Annual SPI RP/C Institute
6 proceedings 1973, Section 3-A) describe the production
7 of wool and fibres, respectively, from molten glass.
8 The glass fibres can be used for insulation,
9 construction or even communication purposes. Glass
10 wool also finds uses in packaging and insulation
11 applications.

12
13 Normally, glass fibres are produced from molten glass
14 using traditional fibre pulling techniques; whereby
15 filaments of high temperature molten glass (850°-
16 1300°C) are formed into strands and stretched over pull
17 rolls before being collected onto a reel.

18
19 Glass wool formation is similar in that the glass is
20 initially melted in a crucible. The crucible has
21 suitable apertures to allow filaments of glass to flow
22 downwards, which are then "blown" into wool using jets
23 of either steam or compressed air. Alternatively,
24 glass wool can be formed using a flame attenuation
25 process, developed by Owens-Corning Fibreglass
26 Corporation circa 1940. In this process molten glass
27 passes through a bushing stage where primary filaments
28 approximately 1 mm wide are formed. The fibres are
29 then aligned into an exact uniformly juxtaposed array,
30 using a fibre guide, into a jet flame issuing from an
31 internal combustion burner. The jet flame causes
32 thinning and lengthening of the fibres before they are
33 collected on a steel mesh belt.

34
35 In both cases, the glass is either supplied in molten
36 form direct from a crucible or from a temperature-

1 gradient furnace.

2

3 Generally, water soluble glasses do not lend themselves
4 to these traditional fibre and wool forming techniques.
5 As an example, US Patent 4,604,097 of Graves et al
6 discloses a water soluble drawn fibre, composed
7 primarily of calcium oxide and phosphorous pentoxide.
8 The fibre produced has a very low tensile strength,
9 compared to fibres spun from non-soluble glass
10 compositions.

11

12 Further, water soluble glasses can also be chemically
13 aggressive when molten, unlike traditional glasses
14 where silicon dioxide is used as the glass former.
15 Additionally, the fibres produced are prone to thermal
16 shock and can suffer from devitrification or
17 crystallisation.

18

19 To combat problems of devitrification and
20 crystallisation, water soluble glass fibres have been
21 previously produced in exacting conditions. Thus, for
22 example, Zimmer et al in WO92/07801 discloses drawing
23 fibres from a water soluble glass composed primarily of
24 phosphorus pentoxide, calcium oxide and iron oxide. In
25 order to keep the viscosity of the glass suitable for
26 drawing, the fibres were drawn at 1200°C. Also as a
27 result of the chemically aggressive nature of the glass
28 at that temperature the glass was pulled in an oxygen
29 rich atmosphere (as high as 80% oxygen by volume).
30 Obviously the commercial production of glass fibres
31 under these high temperature controlled atmospheric
32 conditions is expensive.

33

34 The problems of working with water soluble glass are
35 compounded by the very nature of the glass. Metal
36 oxides of elements such as lead and tellurium have

1 previously been used in glass as additives to affect
2 qualities of the glass; crystallisation temperature,
3 viscosity and density, for example. As a result of
4 environmental concerns and particularly when the
5 glasses are to be used in a biological application
6 these additives must be avoided and replaced by more
7 acceptable alternatives.

8
9 Therefore, it is an object of the present invention to
10 provide environmentally acceptable water soluble glass
11 fibres with suitable mechanical properties, and to
12 produce said fibres under less forcing conditions.

13
14 The present invention provides a method for forming a
15 water-soluble glass fibre and/or glass wool, the method
16 comprising producing a water-soluble glass and heating
17 said glass above its melting point to form molten
18 glass, cooling at least a portion of said molten glass
19 to a pre-selected working temperature and then
20 processing said molten glass having said working
21 temperature into fibres and/or wool.

22
23 Generally, the glass is initially heated to a melting
24 temperature of 500°-1200°C, preferably 750°-1050°C.
25 The temperature is then slowly lowered to the working
26 temperature at which fibre formation occurs.

27
28 Generally, the working temperature of the glass will be
29 at least 200°C lower than the temperature at which the
30 glass is initially heated. Suitable working
31 temperatures may fall within the following ranges 400°-
32 500°C, 500°-900°C (preferably 550°-700°C, more
33 preferably 550°-650°C, especially 600°-650°C) and 800-
34 1000°C. The working temperature selected will depend
35 upon the glass composition, but an approximate
36 indication of a suitable working temperature can be

1 established as hereinafter described. Depending upon
2 the glass composition used, the working temperature may
3 be a range of suitable temperatures. The range of
4 working temperatures may be narrow, for example of only
5 10°C, so that fibre formation may occur only between
6 the temperature of N°C to (N+10)°C. Other glass
7 compositions may have a wider temperature range for the
8 working temperature in which glass formation is
9 possible.

10
11 Alternatively, the working temperature of the glass may
12 be defined as 50-300°C above the Tg of the glass.

13
14 In order to obtain an approximate indication of the
15 working temperature for any particular glass
16 composition, the glass composition should be slowly
17 heated to its melting point. As soon as the glass is
18 molten, frequent attempts to pull the composition
19 upwardly to form a fibre should be made, with the
20 temperature of the composition being very gradually
21 increased between attempts. The temperature range of
22 the composition during which fibre formation is
23 possible should be noted and used as a preliminary
24 working temperature in the process of the invention.

25
26 It will be clear to those skilled in the art that the
27 pulling speed at which the fibre is drawn off can
28 affect the choice of working temperature and the
29 diameter of the fibre required. Where a fibre of
30 relatively large diameter is required, the fibre tends
31 to be pulled more slowly and the working temperature
32 may need to be decreased slightly. Where a fibre of
33 relatively small diameter is required (eg a glass
34 wool), the fibres may be drawn at the much higher
35 pulling speed and the working temperature may need to
36 be increased (thus lowering the viscosity of the

1 composition to accommodate the increased pulling
2 speed). Selection of the exact working temperature in
3 respect of any particular fibre size and composition
4 will be a simple matter of routine evaluation of
5 optimal process conditions.

6
7 With reference to the "working temperature" of the
8 glass, the skilled person will appreciate that the
9 furnace temperature may differ considerably from the
10 temperature of the glass itself and indeed there may be
11 a significant temperature gradient in the glass.
12 Ideally the "working temperature" will be the
13 temperature of the glass as fibre formation (ie.
14 pulling) takes place. In many compositions however, it
15 may not be practical to measure the temperature at the
16 surface of the glass where pulling occurs by insertion
17 of a temperature probe as the introduction of the probe
18 may precipitate crystallisation of the glass. One
19 alternative is to place a temperature probe into the
20 bushing and to monitor the bushing temperature which
21 will be a good indicator of the glass temperature at
22 the moment of fibre formation. Alternatively an Infra
23 Red pyrometer may be focused onto the appropriate area
24 of the glass and used to monitor the temperature.

25
26 The glass to be formed into fibres will generally be
27 heated until molten, optionally clarified, and then
28 cooled slowly and controllably until the appropriate
29 working temperature is reached and fibre formation can
30 commence. The initial heating of the glass above its
31 melting point and the subsequent fibre formation may be
32 carried out in a single vessel or, alternatively, the
33 molten glass may be transferred to a vessel designed
34 specifically for fibre formation. One way of holding
35 the molten glass in a vessel having a bushing within
36 its lower surface until the temperature drops to the

1 required working temperature is to coat or fill the
2 holes of the bushing with a material that gradually
3 melts over the period of time taken for the glass to
4 reach the temperature required.

5
6 The most important aspect of the present invention is
7 the manner in which the working temperature is reached.
8 We have found that the molten glass, which may
9 preferably be heated significantly above its melting
10 point, should be allowed to cool in a highly controlled
11 manner, the temperature being only gradually reduced
12 until the working temperature is reached. A stirrer
13 may be present to ensure that the temperature of the
14 whole of the molten glass is kept as uniform as
15 possible.

16
17 The glass is cooled to a temperature at which the glass
18 will not crystallise for at least the period of time
19 needed to convert the melt to fibre. This temperature
20 is termed a "holding temperature". The rate of cooling
21 from this holding temperature is determined by the rate
22 at which the melt is consumed at the bushing and the
23 difference in temperature between the bushing
24 temperature (the working temperature) and the melt
25 holding temperature.

26
27 Due to low viscosity and narrow temperature band for
28 many of these compositions, control of the balance
29 between melt temperature, bushing temperature and glass
30 throughput rate is critical.

31
32 According to a further aspect of the present invention
33 there is provided a composition suitable for processing
34 into glass fibres and/or wool.

35
36 Phosphorous pentoxide (P_2O_5) is preferably used as the

1 glass former.

2

3 Generally the mole percentage of phosphorous pentoxide
4 in the glass composition is less than 85%, preferably
5 less than 60% and especially between 30-60%.

6

7 Alkali metals, alkaline earth metals and lanthanoid
8 oxides or carbonates are preferably used as glass
9 modifiers.

10

11 Generally, the mole percentage of alkali metals,
12 alkaline earth metals and lanthanoid oxides or
13 carbonates is less than 60%, preferably between 40-60%.

14

15 Boron containing compounds (eg B_2O_3) are preferably used
16 as glass additives.

17

18 Generally, the mole percentage of boron containing
19 compounds is less than 15% or less, preferably less
20 than 5%.

21

22 Other compounds may also be added to the glass to
23 modify its properties, for example SiO_2 , Al_2O_3 , SO_3 ,
24 sulphate ions (SO_4^{2-}) or transition metal compounds (eg.
25 first row transition metal compounds).

26

27 Typically the soluble glasses used in this invention
28 comprise phosphorus pentoxide (P_2O_5) as the principal
29 glass-former, together with any one or more
30 glass-modifying non-toxic materials such as sodium
31 oxide (Na_2O), potassium oxide (K_2O), magnesium oxide
32 (MgO), zinc oxide (ZnO) and calcium oxide (CaO). The
33 rate at which the glass dissolves in fluids is
34 determined by the glass composition, generally by the
35 ratio of glass-modifier to glass-former and by the
36 relative proportions of the glass-modifiers in the

1 glass. By suitable adjustment of the glass
2 composition, the dissolution rates in water at 38°C
3 ranging from substantially zero to 25mg/cm²/hour or more
4 can be designed. However, the most desirable
5 dissolution rate R of the glass is between 0.01 and
6 2.0mg/cm²/hour.

7
8 The water-soluble glass is preferably a phosphate
9 glass, and preferably comprises a source of silver ions
10 which may advantageously be introduced during
11 manufacture as silver orthophosphate (Ag₃PO₄). The
12 glass preferably enables controlled release of silver
13 and other constituents in the glass and the content of
14 these additives can vary in accordance with conditions
15 of use and desired rates of release, the content of
16 silver generally being up to 5 mole %. While we are
17 following convention in describing the composition of
18 the glass in terms of the mole % of oxides, of halides
19 and of sulphate ions, this is not intended to imply
20 that such chemical species are present in the glass nor
21 that they are used for the batch for the preparation of
22 the glass.

23
24 The optimum rate of release of silver ions into an
25 aqueous environment may be selected by circumstances
26 and particularly by the specific function of the
27 released silver. The invention provides a means of
28 delivering silver ions to an aqueous medium at a rate
29 which will maintain a concentration of silver ions in
30 said aqueous medium of not less than 0.01 parts per
31 million and not greater than 10 parts per million. In
32 some cases, the required rate of release may be such
33 that all of the silver added to the system is released
34 in a short period of hours or days and in other
35 applications it may be that the total silver be
36 released slowly at a substantially uniform rate over a

1 period extending to months or even years. In
2 particular cases there may be additional requirements,
3 for example it may be desirable that no residue remains
4 after the source of the silver ions is exhausted or, in
5 other cases, where the silver is made available it will
6 be desirable that any materials, other than the silver
7 itself, which are simultaneously released should be
8 physiologically harmless. In yet other cases, it may
9 be necessary to ensure that the pH of the resulting
10 solution does not fall outside defined limits.

11
12 Generally, the mole percentage of these additives in
13 the glass is less than 25%, preferably less than 10%.

14
15 Embodiments of the invention will be described with
16 reference to the following non-limiting examples.

17
18 **Example 1**

19	Component	Mole %
20	Glass Composition	
21	Na ₂ O	31.05
22	CaO	16.00
23	Ag ₂ O	3.88
24	P ₂ O ₅	46.08
25	Na ₂ PO ₃ F	0.97
26	2Al ₂ O ₃ .B ₂ O ₃	2.00

27
28 100 grams of the sample was heated to 900°C before
29 being cooled and pulled at 650°C, at 25 km/hr. Overall
30 the fibre was good; one sample was 10 km in length and
31 11 grams in weight, although there was some
32 crystallisation at the pulling temperature.

33

34

1 **Example 2**

2		Component	Mole %
3	Glass Composition		
4		Na ₂ O	29.51
5		CaO	15.21
6		Ag ₂ O	3.68
7		P ₂ O ₅	43.80
8		2Al ₂ O ₃ .B ₂ O ₃	1.90
9		Na ₂ PO ₃ F	1.90
10		Na ₂ B ₄ O ₇ .10H ₂ O	1.00
11		Na ₂ PO ₄	3.00

12

13 74 grams of the sample was heated to 1000°C before
14 being cooled and pulled at 635°C at 25 km/hr. The
15 fibre produced was ultrafine; one sample was 18 km in
16 length and 59 grams in weight. The sample was sprayed
17 with WD40 to prevent water absorption and to aid
18 lubricity. There was some debris at the bottom of the
19 crucible, but this was found to be just iron deposits
20 from the brushing rod.

21

22 **Example 3**

23

24		Component	Mole %
25	Glass Composition		
26		Na ₂ O	34.20
27		CaO	16.15
28		P ₂ O ₅	44.65
29		Na ₂ SO ₄	5.00

30

31 200 grams of the sample was heated to 1050°C before
32 being cooled and pulled at 635°C at 25 km/hr. The
33 fibre was good although there was some crystallisation
34 at the pulling temperature.

35

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1 **Example 4**

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4 **Glass Composition**

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12 117 grams of the sample was heated to 950°C before
13 being cooled and pulled at 635°C, at 40 km/hr. The
14 fibre produced was good and there were no
15 crystallisation problems even though the surface
16 temperature of the fibre dropped to 510°C in the
17 pulling process.

18

19 **Example 5**

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21

22 **Glass Composition**

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30 99 grams of the sample was heated to 800°C before being
31 cooled to 650°C and pulled at 40 km/hr. The fibre
32 produced was very fine but difficult to pull and quite
33 fragile at speed.

34

35

36

Component	Mole %
Na ₂ O	32.40
CaO	15.30
P ₂ O ₅	42.30
2Al ₂ O ₃ .B ₂ O ₃	3.00
Na ₂ PO ₃ F	1.00
Na ₂ SO ₄	6.00

Component	Mole %
Na ₂ O	31.71
CaO	14.73
P ₂ O ₅	36.33
B ₂ O ₃	4.78
SO ₃	9.40
Na ₂ PO ₃ F	3.00

1 **Example 6**

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5 **Glass Composition**

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16 200 grams of the sample was heated to 850°C before
17 being cooled to 545°C and pulled at 40 km/hr. The
18 fibre produced was strong and thin; there was not a
19 problem of crystallisation, in fact the glass can be
20 stored at 550°C for 72 hours without the onset of
21 crystallisation.

22

23 **Example 7**

24 Below is an example of a wool formulation and running
25 conditions to illustrate the differences with the
26 monofilament examples given above.

27

28 A typical wool formulation is

29

30	Na ₂ O	26.31
31	CaO	17.78
32	P ₂ O ₅	47.04
33	B ₂ O ₃	5.94
34	MnO	1.55
35	Fe ₂ O ₃	0.97
36	NaF	0.41

37

38 Solution rate, non annealed = 0.0278 mg.cm⁻²hr⁻¹

1 Melted and refined at 1000°C.
2 Cooled and held at 725°C.
3 Bushing temperature maintained at 365°C.
4
5 Thick fibres approx 1.2mm diameter drawn through pinch
6 rollers at 2.5 M.mm⁻¹ from a bushing with 6 x 6.5mm
7 diameter holes. Fibres jet attenuated to produce a
8 fine wool 5 -15µm diameter. The wool was sprayed with
9 silicone oil finish during the attenuation process and
10 collected on a stainless steel mesh conveyor.
11 Typically, attenuated wools will have diameters of 5 to
12 20µm. Monofilament fibres will mostly be 20 to 50µm
13 diameter.

14
15 **Example 8**

16	Na ₂ O	31.19 mole %
17	K ₂ O	9.63 mole %
18	Ag ₂ O	2.9 mole %
19	B ₂ O ₃	2.74 mole %
20	2NaF	0.66 mole %
21	P ₂ O ₅	52.88 mole %

22 Furnace at 710°C - 800°C.
23 Bushing at 450°C - 460°C.
24 4.5mm bushing holes.
25 50km per hour pull rate.
26 Good fibres.
27 Solution rate = 1.68 not annealed 2.28 annealed.

28
29 **Example 9**

30	Na ₂ O	32 mole %
31	K ₂ O	10 mole %
32	Ag ₂ O	3 mole %
33	P ₂ O ₅	55 mole %

34

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1 Furnace at 850°C.
2 Bushing at 530°C.
3 5mm bushing holes.
4 55kmph.
5 Good strong fibres.

6
7 **Example 10**

8	Na ₂ O	32 mole %
9	K ₂ O	10 mole %
10	(MgO	4 mole %) - added as an anti-microbial
11	B ₂ O ₃	5 mole %
12	Ag ₂ O	3 mole %
13	P ₂ O ₅	46 mole %

14
15 Furnace temperature 650°C - 730°C.
16 Bushing temperature 410°C - 420°C.
17 Bushing 5.5mm diameter.
18 Speed up to 100kmph.
19 Solution rate 0.7 annealed 1.0 non annealed (mg.cm⁻³.hr⁻¹).
20 Very good strong reliable fibre. Very stable.

21
22 **Example 11**

23	Na ₂ O	36.68 mole %
24	K ₂ O	8.63 mole %
25	P ₂ O ₅	45.09 mole %
26	B ₂ O ₃	5.29 mole %
27	Ag ₂ O	2.59 mole %
28	(CaO	1.73 mole % to attenuate solution rate!

29
30 Furnace temperature 550°C.
31 Bushing 62 x 5.0mm holes.
32 Bushing temperature 400°C.
33 Speed 80kmph.
34 Very good fibres.
35 Solution rate 3.11 annealed, 3.8 non annealed (mg.cm⁻².hr⁻¹).
36
37 The fibres show excellent tensile strength, flexibility
38 and shock resistance.

1 The fibres are especially suitable for industrial and
2 plastics reinforcement controlled release (anti-
3 microbial, anti-corrosion etc) and rapidly
4 biodegradable applications.
5

6 **Example 12**

7	CaO	30 mole %
8	MgO	20 mole %
9	P ₂ O ₅	50 mole %

10

11 Furnace at 1050°C.

12 Bushing 5.5mm holes.

13 Bushing temperature 700°C - 720°C.

14 Speed up to 80kmph.

15 Solution rate TBA.

16 Very strong fibre.

17

18 **Example 13**

19	(K ₂ O	5 mole %)	Trace to alter dissolution rate
20	CaO	25 mole %	
21	Mg ₂ O	20 mole %	
22	P ₂ O ₅	50 mole %	

23

24 Furnace 1000°C.

25 Bushing 5.5mm.

26 Bushing temperature 560°C - 620°C.

27 Speed up to 70kmph.

28 Solution rate TBA.

29 Very strong fibre.

30 Anti-microbial.

31

32 **Example 14**

33	CaO	28.5 mole %
34	MgO	18.5 mole %
35	Ag ₂ O	3 mole %
36	P ₂ O ₅	50 mole %

37

38

- 1 Furnace temperature 1050°C - 1150°C.
- 2 Bushing 4 x 5.5mm.
- 3 Bushing temperature 700°C.
- 4 Speed 50kmph.
- 5 Solution rate TBA.
- 6 Very good, strong fibre.
- 7 Anti-microbial.

8

9 **Example 15**

- | | | |
|----|-------------------------------|-----------|
| 10 | CaO | 30 mole % |
| 11 | MgO | 20 mole % |
| 12 | P ₂ O ₅ | 50 mole % |

13

- 14 As Example 14 (without silver)

15

- 16 The fibres show excellent tensile strength, flexibility
17 and shock resistance. These fibres are suitable for
18 applications requiring slower release and greater
19 tensile strength plus biodegradability. The fibres are
20 suitable for orthopaedic implants and tissue
21 engineering applications.

22

23

24

1 **Claims**

- 2
- 3 1. A method for forming water-soluble glass fibres,
4 the method comprising providing a composition
5 suitable for producing a water-soluble glass and
6 heating said composition above the melting point
7 of said glass to form a molten glass, cooling at
8 least a portion of said molten glass to a pre-
9 selected working temperature and then processing
10 said molten glass having said working temperature
11 into fibres.
- 12
- 13 2. A method as claimed in Claim 1 wherein said
14 portion of said molten glass is cooled slowly to
15 said working temperature.
- 16
- 17 3. A method as claimed in either one of Claims 1 and
18 2 wherein said working temperature is 50-300°C
19 above the Tg of the glass.
- 20
- 21 4. A method as claimed in either one of Claims 1 and
22 2 wherein said working temperature is at least
23 200°C below the temperature to which the glass is
24 initially heated.
- 25
- 26 5. A method as claimed in any one of Claims 1 to 4
27 wherein glass wool is formed.
- 28
- 29 6. A method as claimed in any one of Claims 1 to 5
30 wherein phosphorous pentoxide is used as the glass
31 former.
- 32
- 33 7. A method as claimed in any one of Claim 6 wherein
34 boron containing compounds are used as glass
35 modifiers.
- 36
- 37 8. A method as claimed in Claim 7 wherein B₂O₃ is used
38 as a glass modifier at a mole percentage of 15% or

1 less.

2

3 9. A method as claimed in any one of Claims 1 to 8
4 wherein said glass is a silver-ion releasing
5 glass.

6

7 10. A method as claimed in Claim 9 wherein silver
8 orthophosphate is added during manufacture of the
9 glass as a source of silver ions.

10

11

INTERNATIONAL SEARCH REPORT

International Application No.

PCT/GB 98/01597

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C03C13/00 C03B37/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C03B C03C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 578 023 A (CORNING INC) 12 January 1994 see page 4, line 20 - page 5, line 34; claim 4; tables I-III ---	1-6
A	WO 86 04807 A (UNIVERSITY OF DAYTON, US) 28 August 1986 cited in the application see claims 1-5 ---	1
A	WO 92 07801 A (ZIMMER INC) 14 May 1992 cited in the application see claims 1,7,8 --- -/--	1



Further documents are listed in the continuation of box C.



Patent family members are listed in annex.

" Special categories of cited documents :

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INTERNATIONAL SEARCH REPORT

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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	<p>DATABASE WPI Section Ch, Week 08 Derwent Publications Ltd., London, GB; Class D22, AN 83-18194K XP002075169 & JP 58 004 821 A (MITSUBISHI MINING & CEMENT CO) , 12 January 1983 see abstract</p> <p style="text-align: center;">---</p>	1
A	<p>DATABASE WPI Section Ch, Week 25 Derwent Publications Ltd., London, GB; Class L01, AN 73-80941U XP002075170 & JP 48 042 814 B (NIPPON SHEET GLASS CO LTD) , 1973 see abstract</p> <p style="text-align: center;">-----</p>	

INTERNATIONAL SEARCH REPORT

Information on patent family members

In International Application No

PCT/GB 98/01597

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